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**CORRELATION OF THE HIGH TEMPERATURE
CREEP CHARACTERISTICS OF CUPRO-NICKEL TO THE
ZENNER HOLMAN PARAMETER**

Richard Lindsay Alford

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CORRELATION OF THE HIGH TEMPERATURE CREEP CHARACTERISTICS OF
CUPRO-NICKEL TO THE ZENNER HOLLOMAN PARAMETER

Richard L. Alford

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ALFORD

1956

THESIS
#373

Letter on cover:

CORRELATION OF THE HIGH
TEMPERATURE CRYSTALLOGRAPHIC
OF CUPRO-NICKEL TO THE ZENER
HOLLOWAY FARM-TER

Richard Lindsay Alford

CORRELATION OF THE HIGH TEMPERATURE CREEP CHARACTERISTICS OF
CUPRO-NICKEL TO THE ZENNER HOLLOMAN PARAMETER

by

Richard Lindsay Alford
Lieutenant, United States Navy

Submitted in partial fulfillment
of the requirements
for the degree of
MASTER OF SCIENCE

United States Naval Postgraduate School
Monterey, California

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This work is accepted as fulfilling
the thesis requirements for the degree of
MASTER OF SCIENCE

from the
United States Naval Postgraduate School

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ABSTRACT

Other experimental investigations have shown that the high temperature creep characteristics of many alloys may be correlated by means of the equation: $\dot{\epsilon} = S e^{-\Delta H/RT} e^{\beta\sigma}$

The results of this work indicate that the activation energy, ΔH , of Cupro Nickel is apparently not a single-valued function of the material but appears to vary with stress and possibly temperature within the ranges investigated i.e.

Stress: 30,000 psi to 35,000 psi and Temperature: 350°C to 450°C.

INTRODUCTION

The high stress and temperatures imposed upon metals as a result of recent developments in power engineering require a new view point to be taken concerning the physical characteristics of metals. Metals can no longer be looked upon as elastic rigid bodies, but must be considered as semi-elastic plastic bodies that flow continuously under load. This time dependent deformation of metals under stress is known as "creep".

Numerous mathematic expressions have been presented for the purpose of correlating the creep characteristics of a metal with stress and temperature. Some of these expressions are empirical in nature which, by and large, attempt to separate the creep strain into transient and steady state components. The theoretical developments are based on an activation process, many of which may be evolved from Eyring's Reaction-Rate Equation.⁽¹⁾

Several years ago Zenner and Hollomon suggested that the flow stress of metals might be related to the temperature and strain rate in accord with the functional equation:

$$\sigma \equiv \sigma(\dot{\epsilon} e^{\Delta H/RT}) \quad (1)$$

The Zenner-Hollomon Parameter also contains the significant tacit implication that the energy of activation, ΔH , is sub-

stantially independent of the state of the material. (2)

Dorn and co-workers utilizing the Zener-Hollomon Parameter in conjunction with the Frying Reaction-Rate Equation met with considerable success in correlating the creep characteristics of high purity aluminum and its alloys which necessarily implies that creep is an activation process. (3,4)

They developed the basic equation:

$$\dot{\epsilon} = S e^{-\Delta H/RT} e^{\beta \sigma} \quad (2)$$

where:

$\dot{\epsilon}$ = creep rate (in. in.⁻¹ hr.⁻¹), In this work $\dot{\epsilon}$ refers to the secondary creep rate.

S = a parameter (hr.⁻¹) which is a function of the creep stress or strain and referred to as the structure parameter.

ΔH = activation energy (cal. mol.⁻¹)

R = the gas constant (cal. mol.⁻¹ °K⁻¹)

T = the absolute temperature (°K)

β = constant (in.² lb.⁻¹)

σ = stress (psi)

For constant load (or constant stress) tests the equation reduces to:

$$\dot{\epsilon} = A e^{-\Delta H/RT} \quad (3)$$

where

A = constant (hr.⁻¹)

ΔH = constant independent of temperature over wide ranges of temperature above 0.45 of the melting point in °K. It is also independent of creep

stress, creep strain, grain size, sub-structures developed during creep, small alloying additions, and cold work.

Thus, the activation energy, ΔH , for creep may be determined by evaluating two creep tests at different temperatures, T_1 and T_2 , under the same loads using the relation:

$$\Delta H = (R \ln \dot{\epsilon}_1 / \dot{\epsilon}_2) / \left(\frac{1}{T_2} - \frac{1}{T_1} \right) \quad (4)$$

The purpose of this work is to attempt to correlate the creep characteristics of commercial Cupro-Nickel using the equations developed by Dorn and co-workers for short time creep tests, the shortest of which was nine minutes and the longest of which was two hours. It was the original intention of the author to continue creep curves to normal rupture, however, though neither copper or nickel is considered as being "notch sensitive", Cupro-Nickel does display such a physical phenomenon and specimens ruptured at the gage points after having entered the secondary stage of the creep curve for a relatively short time.

MATERIALS AND PRELIMINARY WORK

Tensile-creep specimens were cut and machined from a 0.100-inch rolled sheet of commercial Cupro-Nickel* obtained from the Revere Copper and Brass Co., New Bedford, Mass. The axis of the specimens was oriented in a direction parallel to the rolling direction of the stock. A one-inch gage length with a reduced width of 0.250" was used. The dimensions of the specimens are shown in Figure 1. Maximum variations of ± 0.0005 " along the reduced section were obtained in machining. The cross sectional area of a specimen's gage length was determined by averaging three measurements taken along the reduced section.

Preliminary to experimental work, the effect of the proposed high temperatures on the structure and surface of the specimens was determined. Although activation energy is supposedly independent of grain size, it was considered necessary to the control of the experiment to obviate the possibility of recrystallization and ensure uniform grain size prior to application of the creep load. A number of specimens were annealed for two hours and twenty-four hours at constant temperatures

*An analysis of the material used in this investigation was not made. The customary nominal composition of Cupro-Nickel¹ is: Cu: 29%, Ni: 33%, Sn: 1.5%, (Fe, Mn, Pb: 1% or less)

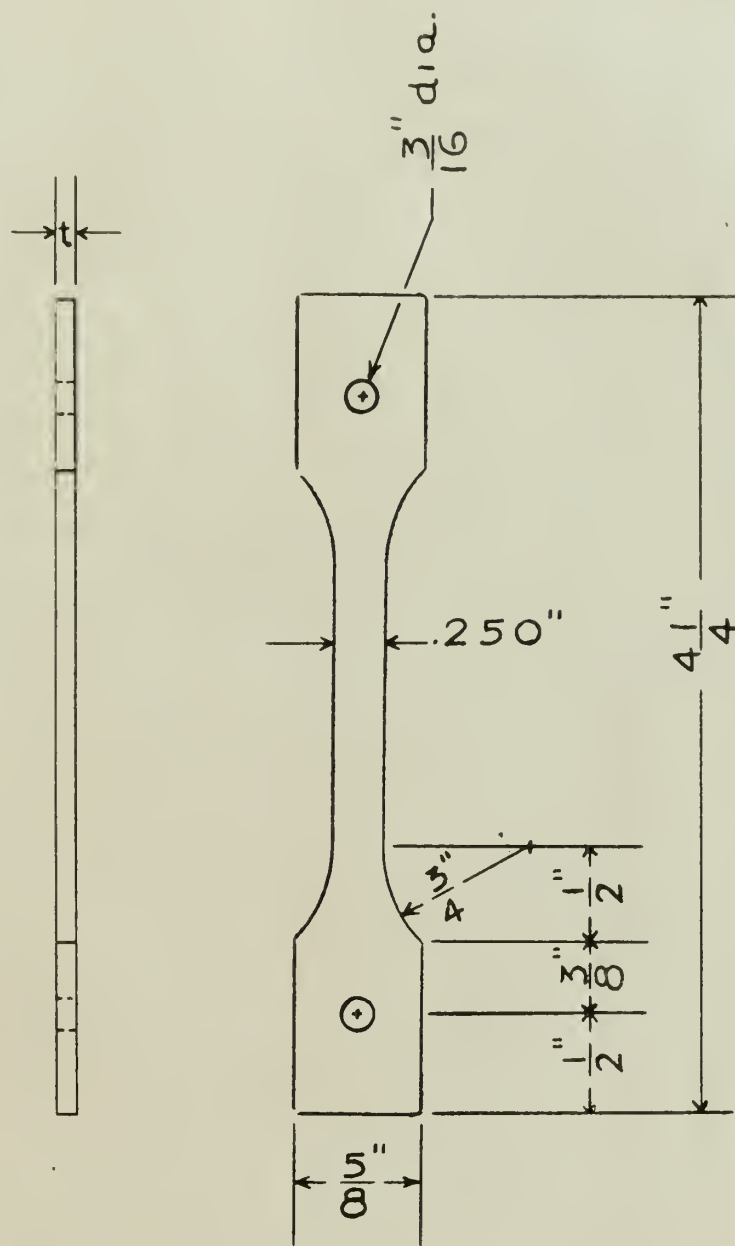


Fig. 1

Dimensions of Tensile Creep Specimen

of 500°C, 600°C, 700°C, and 800°C. Subsequent microscopic examination showed negligible difference in grain size between the annealing times for a given temperature. Surface oxidation was negligible below 500°C. Since the creep tests were to be conducted in a range of temperatures between 350°C and 450°C, it was decided to anneal all creep specimens in a helium atmosphere furnace for a period of two hours at 700°C to obviate the possibility of significant grain growth during creep tests. All creep specimens were furnace cooled. The recrystallized grain size obtained by averaging four linear counts per specimen was 40 grains/mm. Later microscopic examinations of crept specimens revealed no significant change in grain size or structure other than what would be expected to result from deformation sustained during creep tests.

EQUIPMENT AND EXPERIMENTAL TECHNIQUE

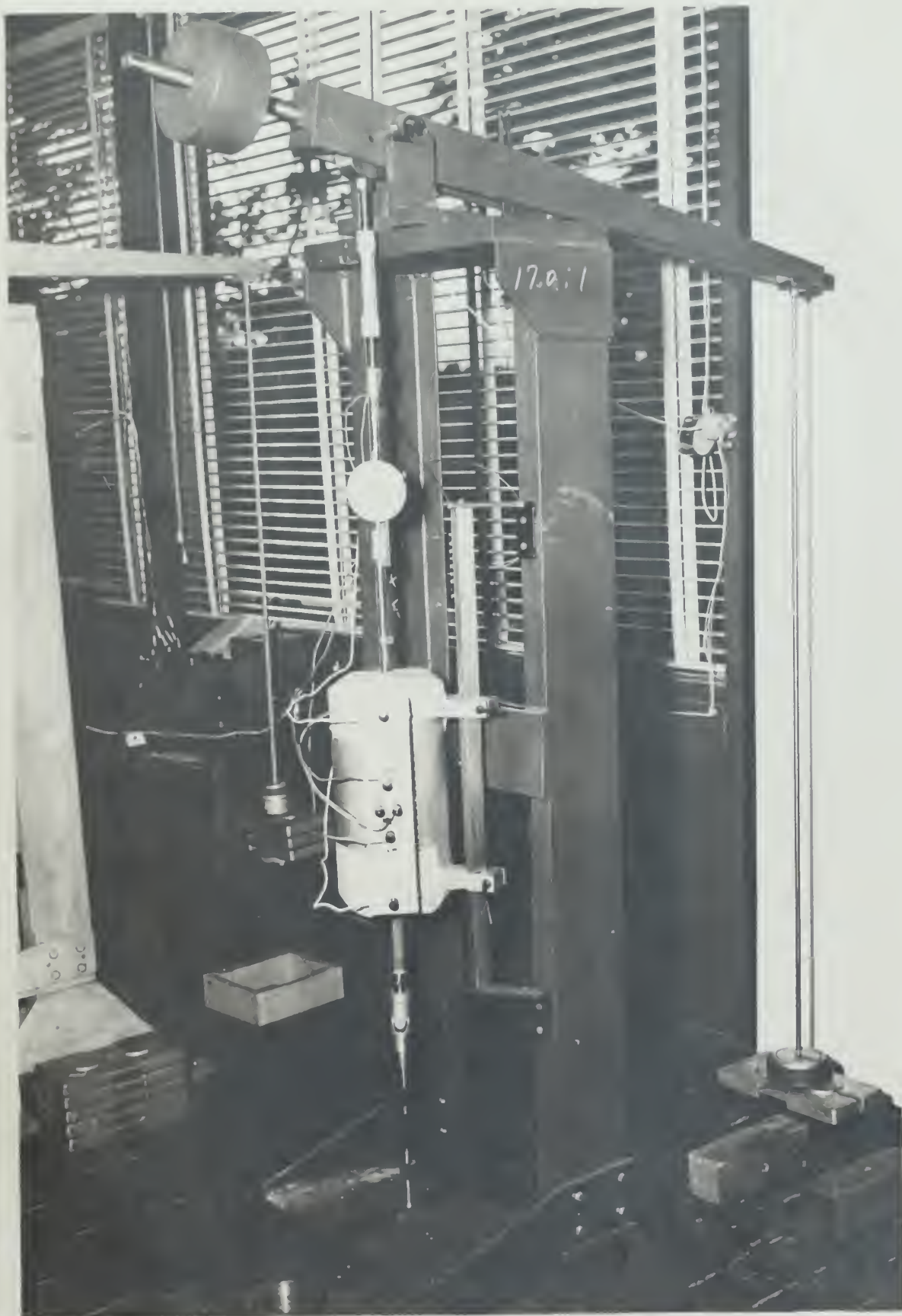
A. Creep Unit (test machine)

The test machine used was a conventional, single lever, constant load type, providing uniaxial loading, (see Figure 2). The test machine lever arm ratio was calibrated with a Baldwin SR-4 Type U (0-2000 lbs) standard load cell which is accurate to 1/4% of full range at a point. The lever arm ratio was found to be 17.00:1. It was demonstrated that the lever gave no detectable loading error within the range of ± 7.5 degrees from the horizontal, thus this lever arm ratio was maintained constant within the limits of the angular displacements developed during the test as a result of specimen extension.

Inasmuch as these experiments are of the constant load type, only the initial stress is of concern. In uniaxial loading the measured cross-sectional area of the specimen is multiplied by the desired initial stress to determine the loading. This load when divided by the mechanical advantage of the lever establishes what weight shall be added to the loading platform. Weights were measured on an accurate scale capable of being read to 0.01 lb.

B. Extensometer Unit

The strain was measured with an extensometer specifically



Creep Unit Figure 2



designed for creep testing at elevated temperatures, (see Figure 3). The features of this unit consist of the following:

- (1) One-inch gage length accurate to $+0.001"$, $-0.000"$ by means of an integral spacer.
- (2) Two gage blocks each containing a carboloy tip which penetrates the specimen $0.010"$ on one side.
- (3) The upper gage block is rigidly connected to a tube which is attached to a dial gage supporting sleeve. The latter slides freely on ball bearings along the upper pull tab of the extensometer unit.
- (4) The lower gage block is rigidly attached to a rod which moves through the above-mentioned tube and provides a resting platform for the dial gage stem which is spring loaded. Additional movement is thus obtained between tube and rod.
- (5) The dial gage has an absolute accuracy of better than $\pm 0.0003"$ with a minimum reading of $0.0001"$.
- (6) All high temperature components of the extensometer were made from type 316 Stainless steel.

C. Temperature Control Unit

An L. H. Marshall 115V nichrome-wound cylindrical type furnace, 12.5" long, 7.0" O.D. and 2.5" I.D. was used. Three individually controlled heating coils were provided in the furnace for temperature gradient control. The coils were connected in series, each coil being in parallel with a variable



Extensometer Unit Figure 3

resistor. Constant voltage supply was obtained from a Sorensen, model 3000-S-115V voltage regulator.* The desired temperature control was obtained by means of a Honeywell "Pulse Pyr-O-Vane" millivoltmeter controller which contained a time-proportioning control. The latter served to decrease the cycling temperature amplitude. The amplitude was further decreased by a resistor placed in parallel with the on and off control breaker. Current in excess of that required to reach temperature was necessary due to variations in room temperature and in insulation around the components protruding from the furnace.

For constant stress, the creep rate varies exponentially with $1/T$. It is mandatory to minimize the temperature gradient across the gage length in order to get accurate data. Because of the nature of the creep equation to be used here, (Equation 3), it is essential to determine the temperature accurately. This was done by means of two duplex fiberglass insulated Chromel-alumel thermocouples attached with glass thread to the specimen just outside of the gage blocks as shown in Figure 3. During the creep tests, the temperature was controlled to better than $\pm 1.5^{\circ}\text{C}$ and a maximum temperature gradient across the gage length of $.25^{\circ}\text{C}$ was maintained. The thermocouple circuit is shown in Figure 4.

*The high instability of line voltage previously resulted in difficulty in obtaining a low cycling temperature amplitude.

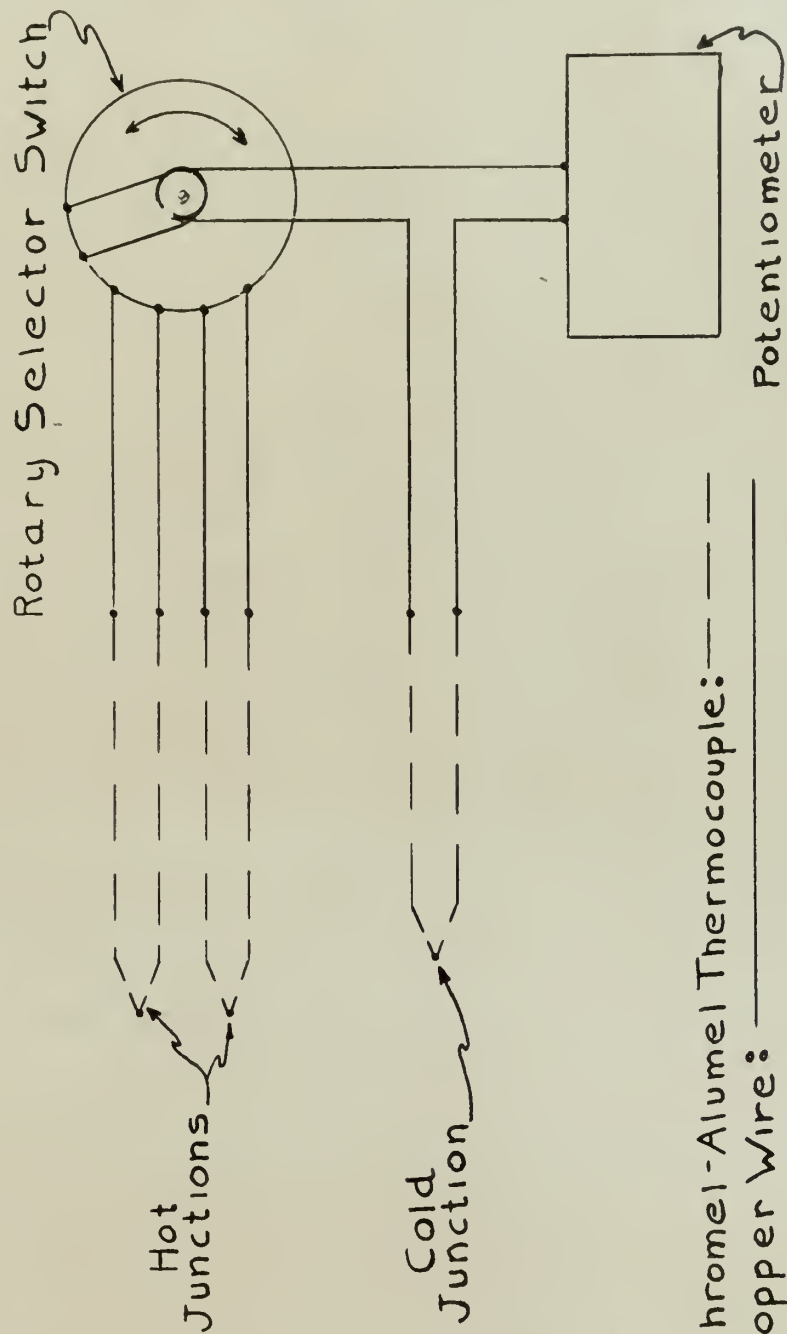


Fig.4
Thermocouple Circuit

D. Test Procedure:

To further obviate the possibility of grain growth and oxidation of the test specimen, while in the creep furnace, the following loading procedure was used to reduce the amount of time during which the creep specimen would be at high temperature:

- (1) A dummy specimen was mounted in the extensometer and thermocouples were attached (see Figure 3).
- (2) The extensometer was placed in the loading system of the test machine and the specimen was centered in the furnace.
- (3) The furnace was sealed top and bottom with three layers of asbestos cloth held in place with masking tape.
- (4) The furnace was heated to test temperature and zero temperature gradient was established across the gage length of the dummy specimen. Five hours were usually required to establish equilibrium.
- (5) The dummy specimen was replaced by a test specimen in the extensometer, thermocouples were reattached, the extensometer was again placed in the test machine with specimen centered in the furnace, and the furnace was resealed.
- (6) Test temperature equilibrium and zero gradient across the gage length were reestablished, this time within one hour.
- (7) As soon as equilibrium was reestablished, the test

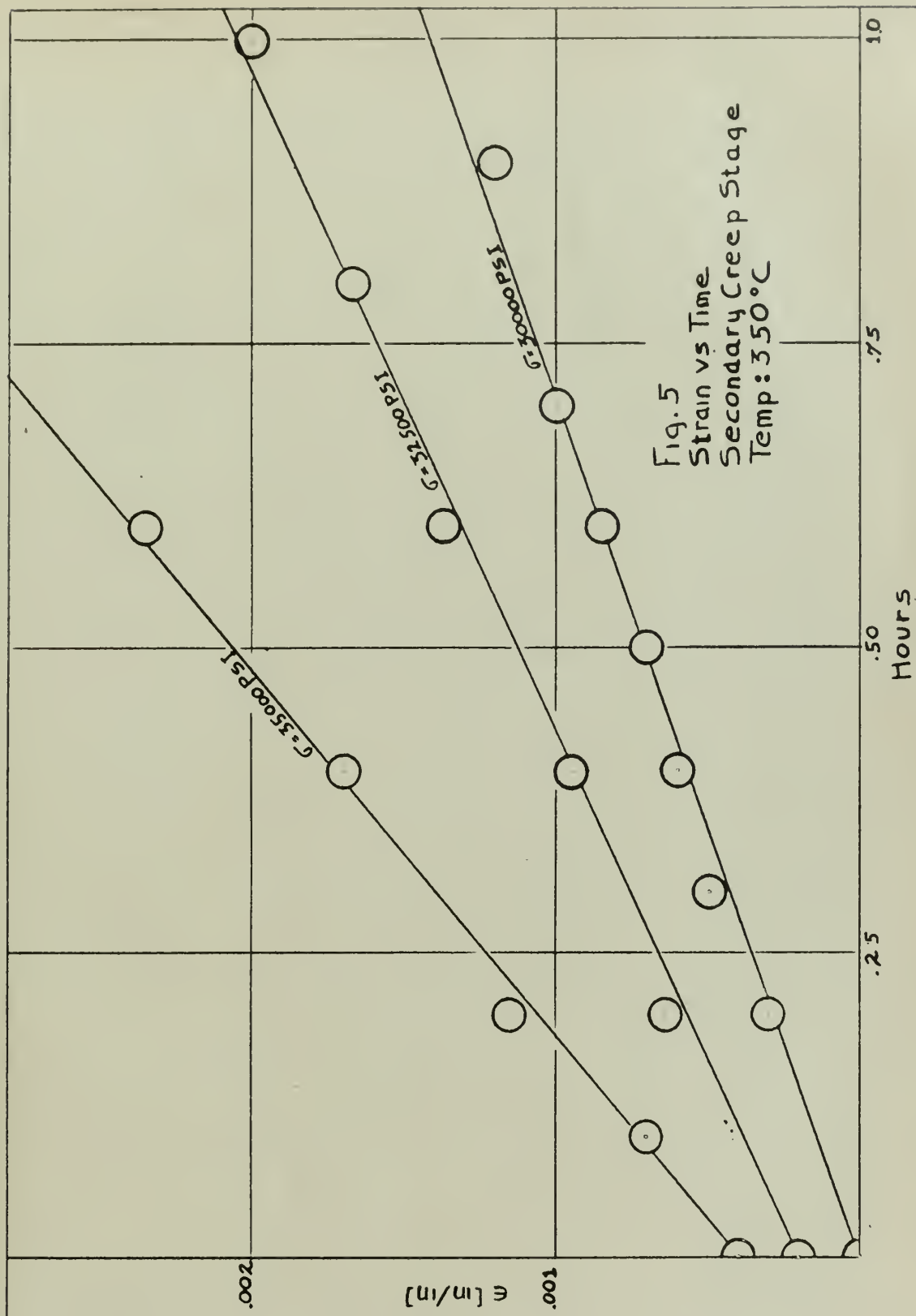
was begun by lowering the weighed load onto the loading platform by means of a screw type automobile jack to reduce dynamic loading as much as possible.

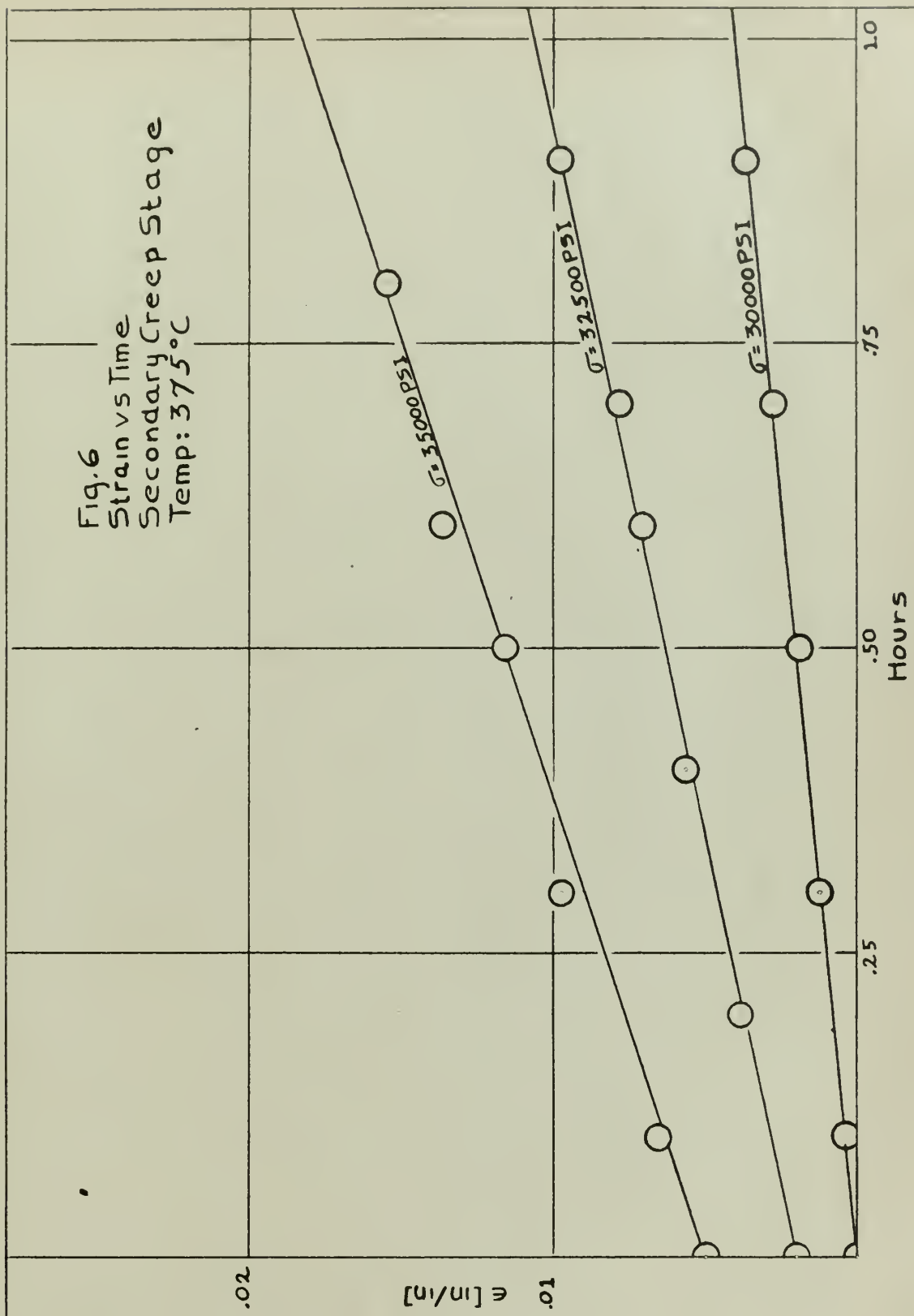
(8) Data was recorded (elongation vs time) until the specimen ruptured at the gage points or until a constant creep rate had been established for at least one hour. The extensometer was read directly to 0.0001" and estimated to 0.00005".

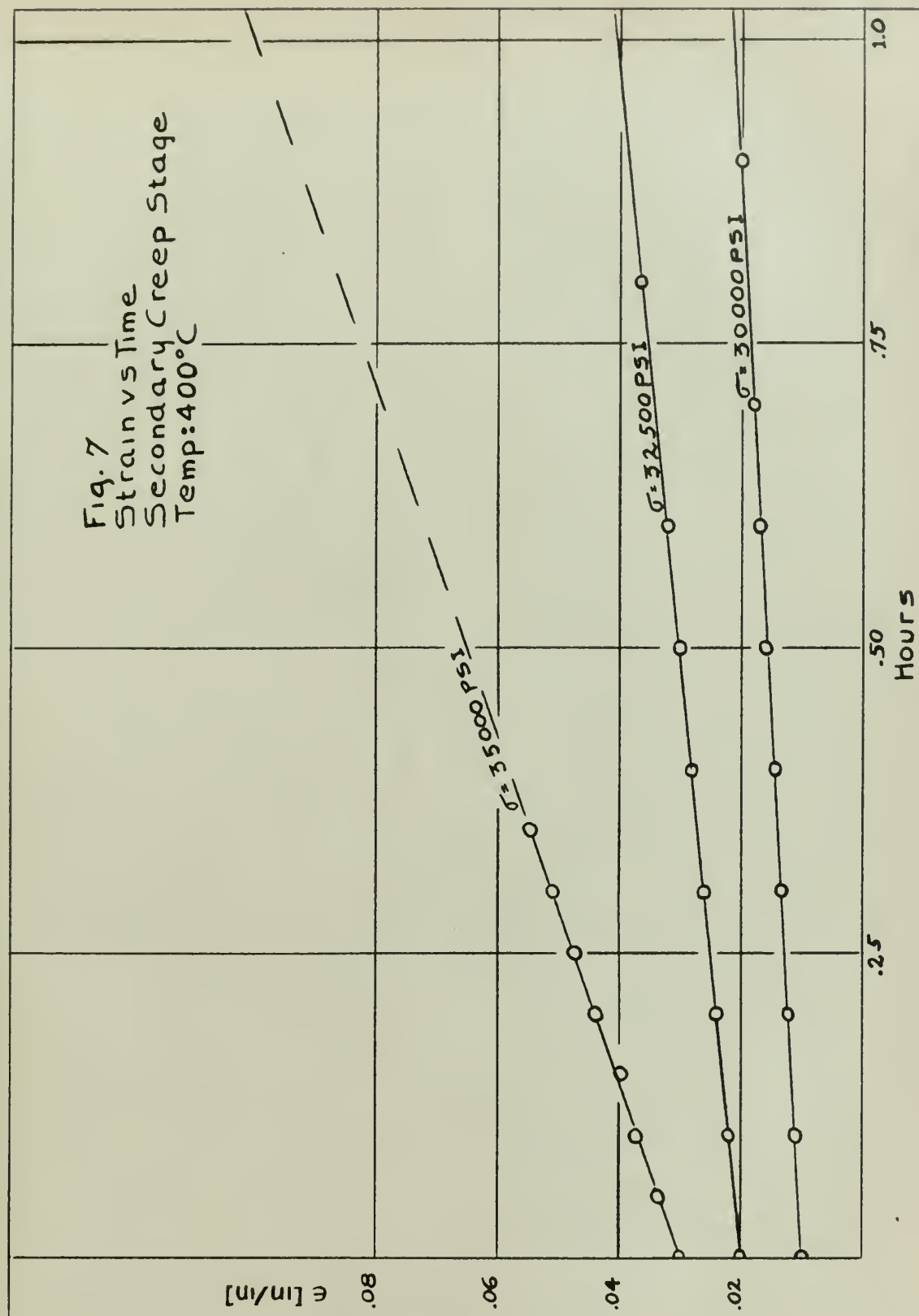
EXPERIMENTAL RESULTS

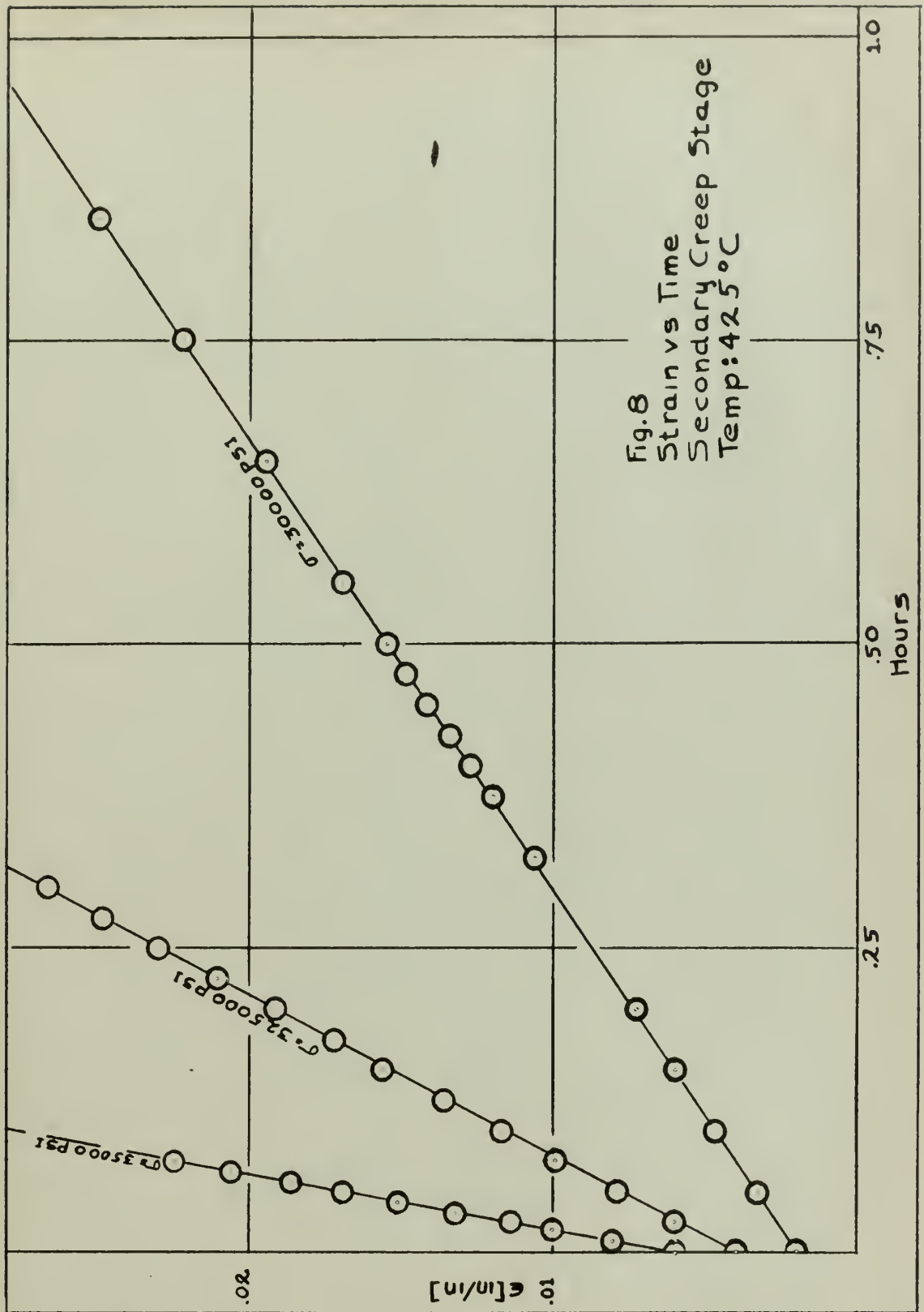
Creep tests were conducted at initial stresses of 30,000 psi, 32,500 psi, and 35,000 psi for each of the following temperatures: 350°C, 375°C, 400°C, 425°C and 450°C. The data recorded during each of these fifteen creep tests is displayed in APPENDIX I. The data of each creep test was plotted as strain vs time and the creep rates were determined from the secondary stages of the creep curves. The extensometer dial gage has an absolute accuracy of ± 0.0003 in. With a minimum dial division of 0.0001 in. and a minimum interpolated reading of .00002 in., the relative accuracy of the readings, with the slack removed from the attendant gear train, may be considered to be better than .00005 in. Under such conditions, creep rates determined over one hour intervals could be expressed with a possible accuracy of $\pm .0001$ in. in.⁻¹ hr.⁻¹. It is to be noted that, due to the scale utilized in Figures 10, 11 and 12 for plotting $\log \dot{\epsilon}$, the intervals of error in creep rate appear, in general, as points. The creep rates determined for each test are recorded at the bottom of the associated test column in APPENDIX I. The secondary creep curves for each test are shown in Figures 5, 6, 7, 8 and 9.

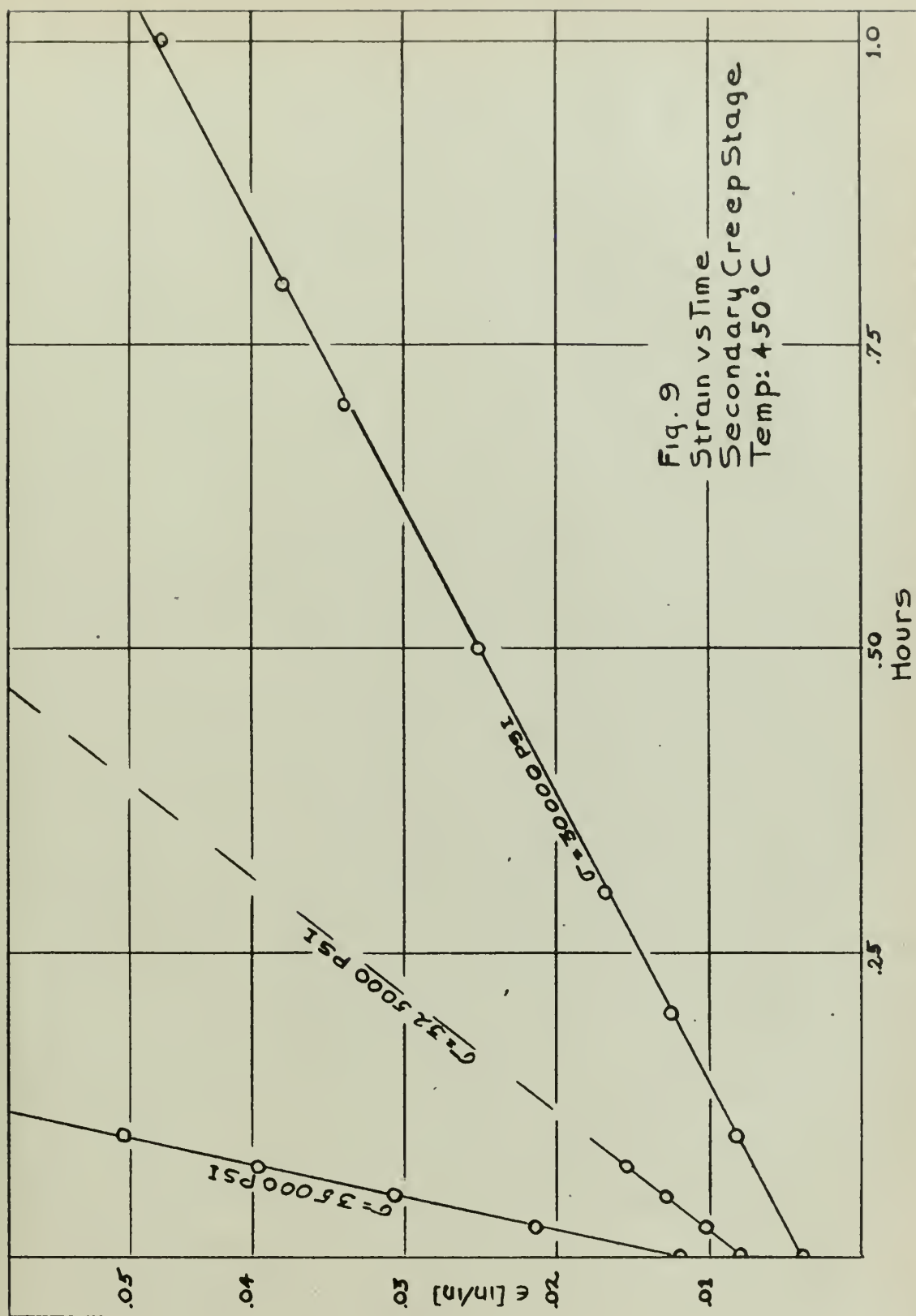
The creep rate data were plotted as $\log \dot{\epsilon}$ vs $1/T$ and regression lines were drawn through points of constant initial





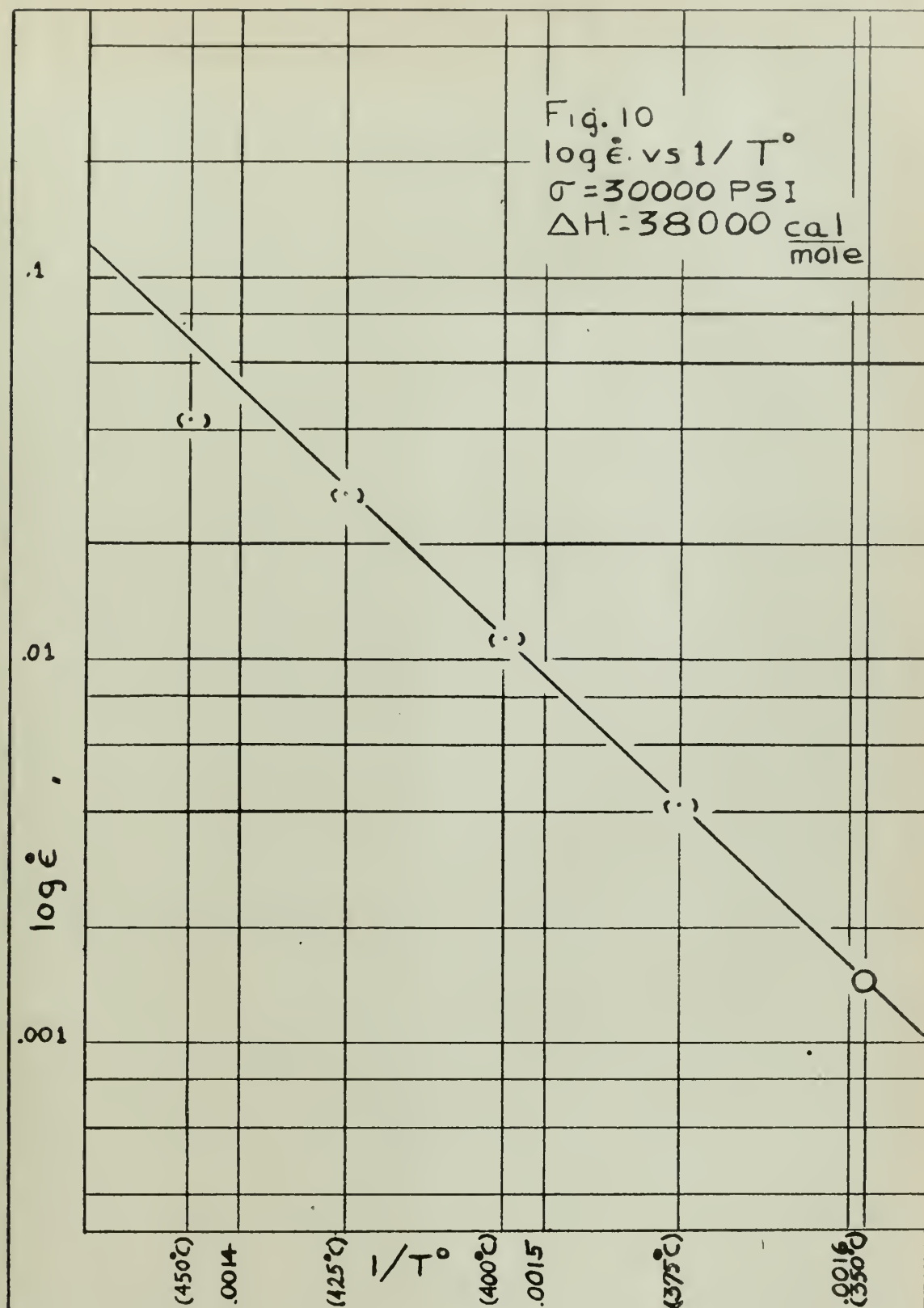


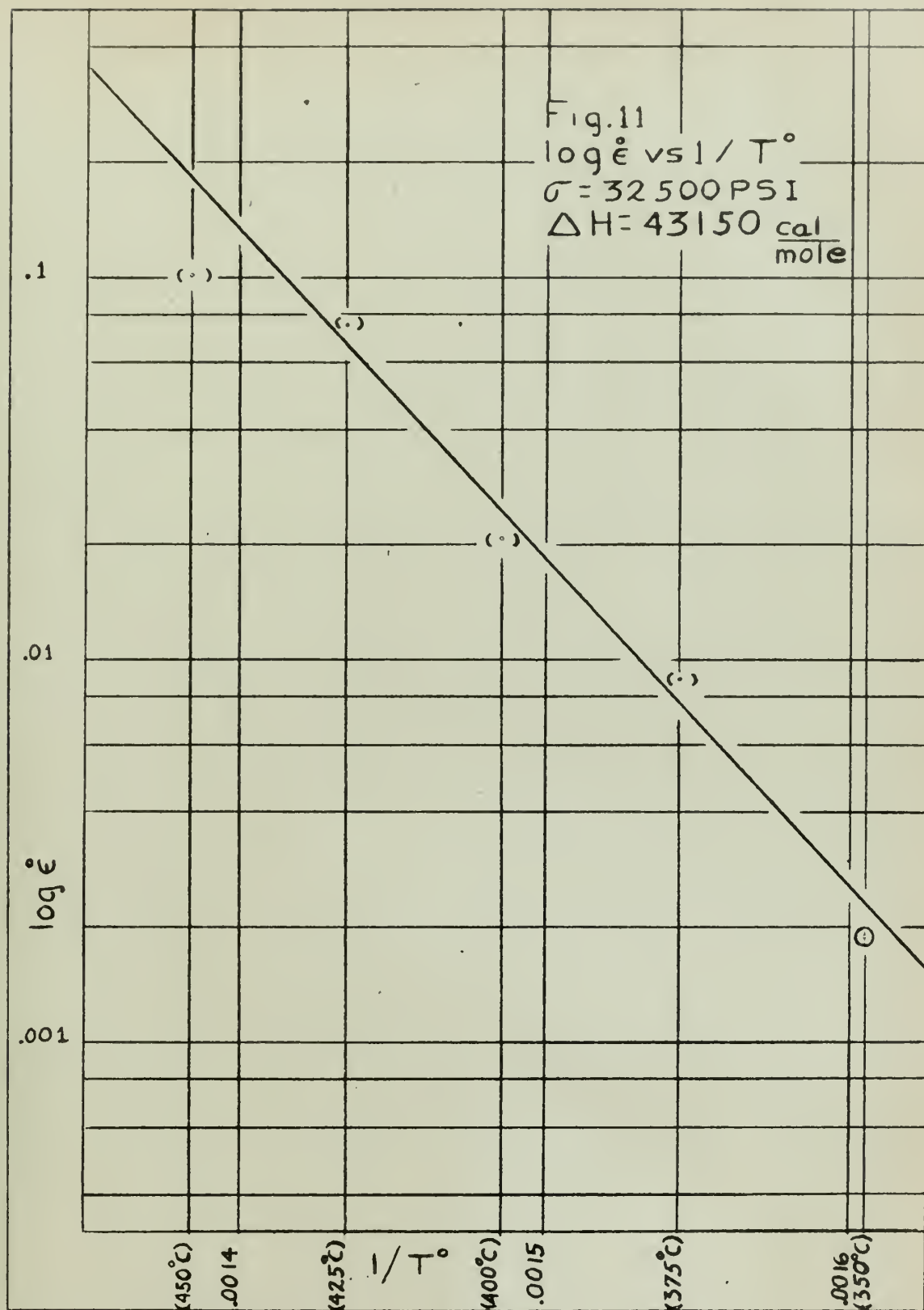


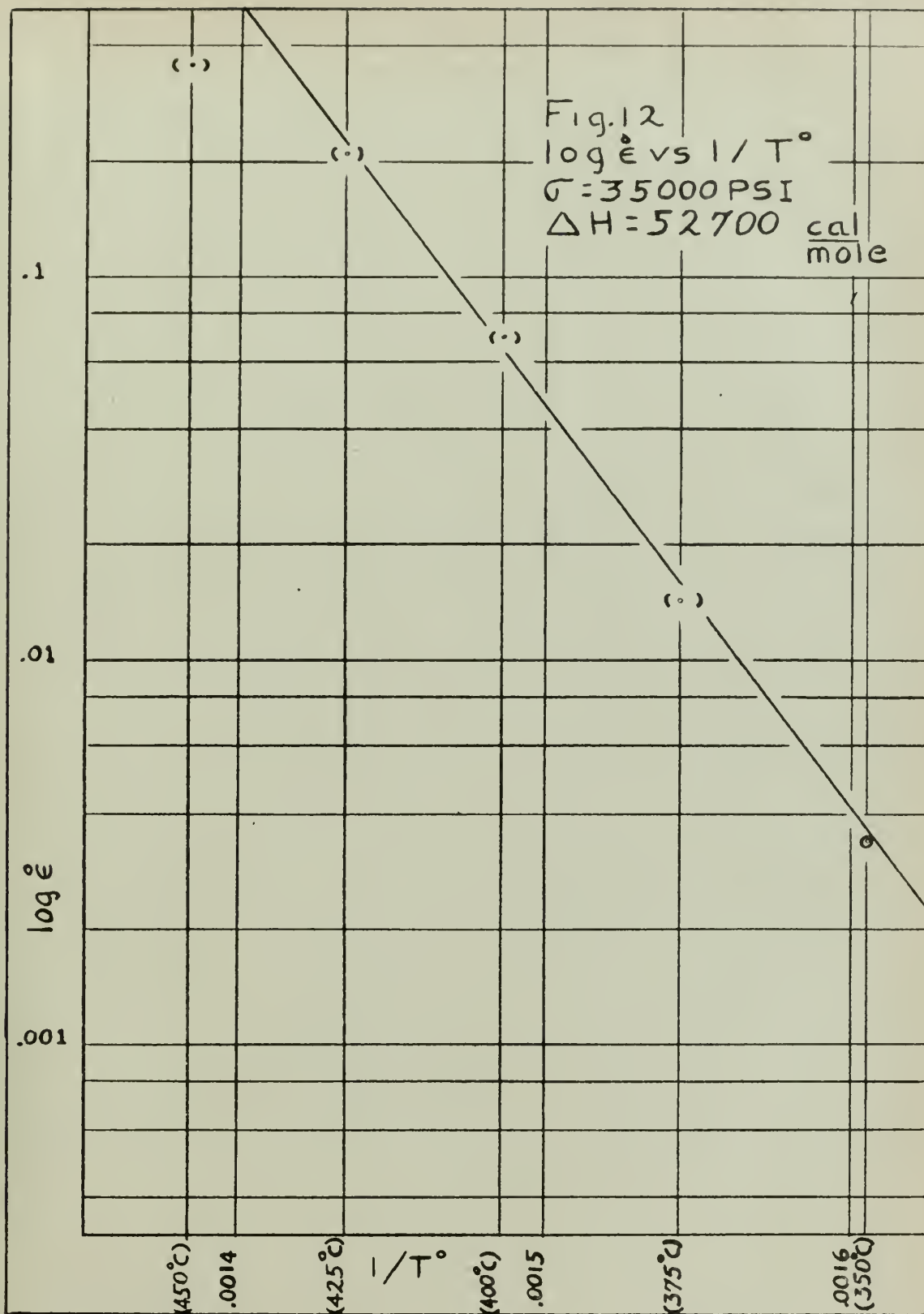


stress, (see Figures 10, 11 and 12). Some fair degree of correlation of the type suggested by Equation 3, between creep rates and constant initial stress was obtained over the range of temperature of 350°C to 425°C.

The consistant manner in which creep rate plots low in relation to the regression lines at a temperature of 450°C suggests the possibility that the creep rate, at higher temperatures, falls off below that predicted by Equation 3. It is to be remembered, that since this work was primarily undertaken with the purpose of determining whether or not creep rates at constant load could be predicted on the basis of Equation 3, which implies a linear correlation, a regression line was thus arbitrarily chosen to be drawn through the points of constant load in Figures 10, 11 and 12. A careful scrutiny of the creep rates as displayed in Figures 10, 11 and 12 discloses that it would not be inconsistent with the data to suggest that a continuous curve, with a slope that decreases with increasing temperature, might be fitted to the data with equal success. This implies that the activation process is not a single one as suggested by a constant ΔH , but rather a complex one consisting of several competing mechanisms each with a unique activation energy. Thus the ΔH is an averaged activation energy which is not constant but varies with temperature depending upon which process is predominant. The data of this work is hardly sufficient upon which to postulate such a possibility, but an interesting trend for further







investigation is believed to be indicated.

The results of the calculations of ΔH using Equation 4 and information obtained from the regression lines shown in Figures 10, 11 and 12 are as follows:

σ (psi)	ΔH
30,000	38,000
32,500	43,150
35,000	52,000

These results indicate that activation energy (ΔH) increases with initial stress and is not therefore a single valued function dependent only on the material as proposed by Dorn and Sherby.

CONCLUSIONS

1. It appears that the creep data of Cupro-Nickel can, to some fair degree, be correlated at a constant initial stress by means of the equation $\dot{\epsilon} = A e^{-\Delta H/RT}$, over a range of temperatures of 350°C to 425°C and a range of initial stresses from 30,000 psi to 35,000 psi. for a relatively short period of time after the completion of the primary or transient stage of the creep curve.
2. A more thorough examination of the data including those obtained at 450°C suggests that ΔH varies with temperature. The apparent constant ΔH , at constant initial stress, in the range of 350°C to 450°C may be fortuitous.
3. ΔH was found to increase from 38,000 cal. mol.⁻¹ to 52,000 cal. mol.⁻¹ with an increase of initial stress from 30,000 psi to 35,000 psi.
4. It is suggested that the activation process in creep is a complex one consisting of several competing mechanisms having some averaged ΔH . The predominant process(es) would depend, among other factors, on stress and temperature. The activation energy is not a single valued function dependent only on the material as proposed by Dorn and Sherby. In this regard, the trend of this work is supported by that of Carreker and Guard. (5)

ACKNOWLEDGEMENTS

It is a pleasure to acknowledge the authors indebtedness to Professor Alfred Goldberg for his invaluable guidance and timely advice, to Professor Frederick L. Coonan and Professor John R. Clark for their interest and advice concerning the project, and to his wife Mrs. R. L. Alford, for her continual cooperation and aid in the compilation of the thesis.

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APPENDIX I
Strain-Time and Strain Rate Data

$C = 30,000 \text{ PSI}$									
$T = 350^{\circ}\text{C}$		$T = 375^{\circ}\text{C}$		$T = 400^{\circ}\text{C}$		$T = 425^{\circ}\text{C}$		$T = 450^{\circ}\text{C}$	
$t(\text{min})$	$\in \times 10^3$	$t(\text{min})$	$\in \times 10^3$	$t(\text{min})$	$\in \times 10^3$	$t(\text{min})$	$\in \times 10^3$	$t(\text{min})$	$\in \times 10^3$
0.0	00.00	0.0	00.00	0.0	00.00	0.0	00.00	0.0	00.00
1.5	13.40	3.0	70.10	3.0	80.50	3.0	60.50	1.5	26.50
4.5	13.70	6.0	70.95	6.0	82.40	6.0	65.80	3.0	30.40
6.0	13.80	9.0	71.50	9.0	83.90	9.0	69.70	4.5	33.35
7.5	13.90	12.0	72.00	10.5	84.50	12.0	72.90	6.0	35.60
9.0	14.00	18.0	72.65	12.0	85.05	18.0	80.70	12.0	44.80
10.5	14.05	24.0	73.30	15.0	86.15	24.0	86.40	18.0	50.40
12.0	14.15	30.0	73.70	18.0	87.30	30.0	89.75	24.0	55.50
18.0	14.40	36.0	74.05	21.0	88.20	33.0	91.40	30.0	60.20
24.0	14.65	48.0	74.90	24.0	89.20	36.0	92.90	36.0	64.60
30.0	14.90	60.0	75.60	27.0	89.95	42.0	94.40	42.0	68.90
42.0	15.20	72.0	76.50	30.0	90.80	48.0	97.30	48.0	73.20
48.0	15.40	84.0	77.40	36.0	92.40	54.0	100.00	54.0	77.35
54.0	15.50	96.0	78.30	42.0	93.75	57.0	101.35	60.0	81.50
60.0	15.60	108.0	79.19	48.0	94.45	60.0	102.70	66.0	85.35
66.0	15.75	120.0	80.00	54.0	95.60	63.0	104.00	72.0	89.30
72.0	15.90			60.0	96.85	66.0	105.30	78.0	94.10
84.0	16.10			66.0	98.00	73.5	108.65	90.0	101.80
				72.0	99.25	76.5	110.10	120.0	122.35
				78.0	100.35	78.0	110.70		
				84.0	101.50	81.0	112.20		
				90.0	102.55	84.0	113.60		
				102.0	104.55	93.0	117.50		
						99.0	122.20		
						104.0	125.00		
$\dot{\epsilon}$		$\dot{\epsilon}$		$\dot{\epsilon}$		$\dot{\epsilon}$		$\dot{\epsilon}$	
.00145		.00420		.01140		.02700		.04215	

C = 32,500 PSI									
T = 350°C		T = 375°C		T = 400°C		T = 425°C		T = 450°C	
t(min)	€ X 10 ³	t(min)	€ X 10 ³	t(min)	€ X 10 ³	t(min)	€ X 10 ³	t(min)	€ X 10 ³
0.0	00.00	0.0	00.00	0.0	00.00	0.0	00.00	0.0	00.00
3.0	87.70	3.0	87.20	3.0	99.00	3.0	107.80	1.5	50.30
6.0	88.15	6.0	88.35	6.0	102.00	6.0	116.40	3.0	53.20
9.0	88.60	12.0	90.10	9.0	104.15	9.0	122.95	4.5	55.50
12.0	88.90	18.0	91.55	12.0	106.00	12.0	128.10	6.0	58.10
15.0	89.20	24.0	92.90	15.0	107.70	15.0	132.70	7.5	60.70
27.0	90.05	30.0	94.05	18.0	109.45	16.5	134.85		
33.0	90.35	36.0	94.95	21.0	110.90	18.0	136.85		
45.0	91.00	42.0	95.90	24.0	112.40	19.5	138.80		
57.0	91.44	48.0	96.85	27.0	113.70	22.0	140.80		
69.0	91.75	54.0	97.70	30.0	114.95	23.5	142.70		
81.0	92.17	60.0	98.55	33.0	116.25	25.0	144.50		
93.0	92.47	66.0	99.39	36.0	117.40	26.5	146.45		
105.0	92.80	72.0	100.10	39.0	118.55	28.0	148.10		
		84.0	102.10	42.0	119.85	29.5	150.05		
				48.0	122.05	31.0	151.95		
				54.0	124.10	32.5	153.90		
				60.0	126.10	34.0	155.70		
				66.0	128.10	35.5	157.50		
				72.0	130.05	37.0	159.35		
				78.0	132.05	38.2	161.20		
				84.0	134.05	40.0	163.20		
				96.0	139.10	43.0	166.90		
						44.5	168.80		
						46.0	170.85		
€	€	€	€	€	€	€	€	€	€
.00190	.00890	.02100	.07520	.10080					

$\sigma = 35,000 \text{ PSI}$									
T = 350 C		T = 375 C		T = 400 C		T = 425 C		T = 450 C	
t(min)	$\epsilon \times 10^3$	t(min)	$\epsilon \times 10^3$	t(min)	$\epsilon \times 10^3$	t(min)	$\epsilon \times 10^3$	t(min)	$\epsilon \times 10^3$
0.0	00.00	0.0	00.00	0.0	00.00	0.0	00.00	0.0	00.00
1.5	32.70	3.0	113.20	3.0	146.10	3.0	121.85	1.5	78.70
3.0	32.90	6.0	115.20	6.0	152.40	4.5	129.35	3.0	91.70
4.5	33.15	9.0	116.85	9.0	157.25	6.0	136.30	4.5	102.10
6.0	33.40	12.0	118.35	12.0	161.50	6.5	138.85	6.0	111.65
7.5	33.60	15.0	119.70	15.0	165.45	7.0	140.45	7.5	120.85
12.0	34.05	18.0	120.85	18.0	169.05	7.5	142.40	9.0	129.70
18.0	34.60	21.0	122.10	21.0	172.70	8.0	144.40	10.5	138.50
24.0	35.10	24.0	123.10	22.5	174.45	8.5	146.30	12.0	147.50
30.0	35.60	27.0	124.10	24.0	176.25	9.0	148.25		
36.0	35.90	30.0	125.00	25.5	177.90	9.5	150.20		
42.0	36.35	33.0	125.80	27.0	179.60	10.0	152.15		
48.0	36.70	36.0	126.55	28.5	181.20	10.5	154.05		
54.0	36.90	39.0	127.30	30.0	182.90	11.0	155.80		
66.0	37.65	42.0	128.05	33.0	186.25	11.5	157.60		
72.0	38.10	45.0	128.80	36.0	189.80	12.0	159.45		
78.0	38.30	48.0	129.75	39.0	193.60	12.5	161.20		
84.0	38.65	54.0	130.95	42.0	197.50	13.0	163.10		
90.0	38.95	57.0	131.65			13.5	164.85		
		66.0	133.70			14.0	166.70		
		78.0	135.50			14.5	168.60		
						15.0	170.50		
$\dot{\epsilon}$	$\dot{\epsilon}$	$\dot{\epsilon}$	$\dot{\epsilon}$	$\dot{\epsilon}$	$\dot{\epsilon}$	$\dot{\epsilon}$	$\dot{\epsilon}$	$\dot{\epsilon}$	$\dot{\epsilon}$
.00340	.01460	.07200	.21000	.35800					

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